



1075280 - R8 SDMS

EMSL ANALYTICAL, INC.
107 HADDON AVENUE
WESTMONT, NJ 08108
PHONE: (845) 469-8671
FAX: (845) 231-6017

Sample Preparation of Libby Duff

ROUND 1

Duff test samples received in zip-lock plastic bags

Sample 1

Initial Weight: 184.97g

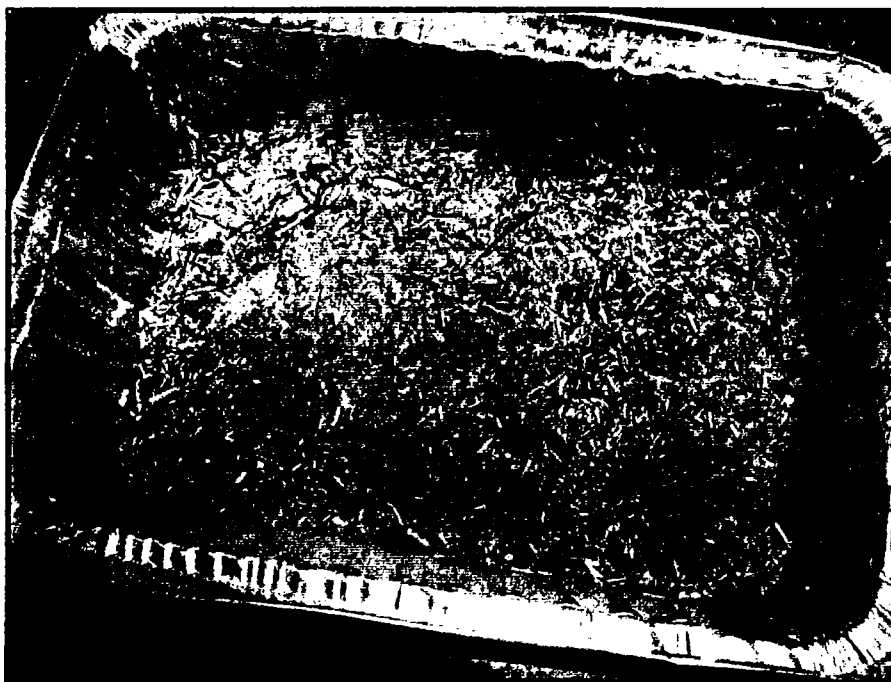
Dry weight: 174.29g

The initial trial involved an incremental ramp and soak scenario as follows:

100 C for 1 hour:	no visible change in sample
200 C for 1 hour:	no visible change
300 C for 3 hours:	smoking, core color now black
400 C for 2 hours:	smoking stopped, sample color black
450 C for 18 hours:	

Results

- Approximately 50% reduction in both volume and mass.
- Some complete ashing but much of the sample is still intact and black (not grey)





1075280 - R8 SDMS

EMSL ANALYTICAL, INC.
107 HADDON AVENUE
WESTMONT, NJ 08108
PHONE: (845) 469-8671
FAX: (845) 231-6017

Sample Preparation of Libby Duff

ROUND 1

Duff test samples received in zip-lock plastic bags

Sample 1

Initial Weight: 184.97g

Dry weight: 174.29g

The initial trial involved an incremental ramp and soak scenario as follows:

- | | |
|---------------------|-------------------------------------|
| 100 C for 1 hour: | no visible change in sample |
| 200 C for 1 hour: | no visible change |
| 300 C for 3 hours: | smoking, core color now black |
| 400 C for 2 hours | smoking stopped, sample color black |
| 450 C for 18 hours: | |

Results

- Approximately 50% reduction in both volume and mass.
- Some complete ashing but much of the sample is still intact and black (not grey)





EMSL ANALYTICAL, INC.
107 HADDON AVENUE
WESTMONT, NJ 08108
PHONE: (845) 469-8671
FAX: (845) 231-6017

ROUND 2

In an effort to increase ashing efficiency of the large duff samples EMSL split the second test sample into 5 individual large crucibles with lids.

A quick ramp to 450 C followed by 18 hours

Results

- Approximately 44% reduction in mass.
- More complete ashing than the first run.
- But still intact structure.



*Decreased
smoking
↳*

The sample was then split

- 1 fraction going to further ashing (another 18 hours of ashing yielded only a 2% reduction)
- 1 fraction going to sonication and filtration

Sonication and Filtration

- 10g of ash put into 1L solution with DI water
- Sonication for 30 minutes
- Filtered 10ml, 25ml and 75ml (all overloaded)
- Lots of debris floats and sinks anyway
- Sieved remainder of unfiltered solution through a 500 micron sieve (lots of debris)

Conclusion: Further matrix reduction necessary (mortar/pestle? Acid?)





EMSL ANALYTICAL, INC.
107 HADDON AVENUE
WESTMONT, NJ 08108
PHONE: (845) 469-8671
FAX: (845) 231-6017

ROUND 3

One of the live duff samples was sieved through a 2mm sieve



Actual samples (not test samples) were received at the lab.

Many were of quite different makeup than the test samples.

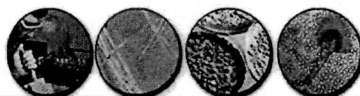
Many were fairly wet and with a fair amount of soil included.

This is sample P1-00089
Station ID: SL 135-06

To estimate the volume of the soil component the sample was dried and sieved through a 2 mm sieve.

The top pan contains the
> 2mm fraction (471.87g)

The bottom pan contains the
< 2mm fraction (232.47)





EMSL ANALYTICAL, INC.
107 HADDON AVENUE
WESTMONT, NJ 08108
PHONE: (845) 469-8671
FAX: (845) 231-6017

A sub-sample of each fraction was gravimetrically reduced by ashing for 18 hours

41.01 % remaining of >2mm fraction -

82.41 % remaining of <2 mm fraction - PLMVE - no milling →

Call notes 10/16/07 0900

1) First and most critical is can we get filterable samples
using acid, mortar & Pestal or Sizermill. If not sieving
makes little sense.

1) week

